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Personal Monitor for Nitrogen Dioxide

Louisiana State Univ, Baton Rouge Dept of Chemistry

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An attempt was made to develop a personal monitor to measure nitrogen dioxide. Sampling of nitrogen dioxide is accomplished by permeation through a silicone membrane into a alkaline thymol blue solution. The nitrogen dioxide is converted to nitrite and is then quantitated by colorimetric analysis. Since collection of the nitrogen dioxide through the silicone membrane depends only on permeation, maintenance of carefully measured sampling rates are not required. A small scale field evaluation of this method with the accepted EPA-TGS-ANSA equivalent method for NO_2 showed that the personal monitor gave much higher results. This difference must be resolved before this personal monitor can be considered as a viable equivalent method for nitrogen dioxide.

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PERSONAL MONITOR FOR NITROGEN DIOXIDE

By

Philip W. West and Kenneth D. Reiszner
Louisiana State University
and Agricultural and Mechanical College
Chemistry Department
Baton Rouge, Louisiana 70803

Grant No. 803193-01

Project Officer

Eva Wittgenstein
Atmospheric Chemistry and Physics Division
Environmental Sciences Research Laboratory
Research Triangle Park, North Carolina 27711

ENVIRONMENTAL SCIENCES RESEARCH LABORATORY
OFFICE OF RESEARCH AND MONITORING
U.S. ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NORTH CAROLINA 27711

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ABSTRACT

An attempt has been made to develop a personal monitor for assaying nitrogen dioxide in ambient air. NO_2 is collected from the ambient air by permeation through a silicone membrane into a alkaline thymol blue solution. The NO_2 is converted to nitrite and determined colorimetrically. Maintenance of a carefully controlled flow rate is not required because the rate of absorption is controlled by permeation. The monitor which is sealed may be worn in any orientation that does not restrict free air movement to the membrane.

Although it is sensitive to ambient air concentrations of nitrogen dioxide and exhibits essentially linear response to this pollutant up to the OSHA limit of 10,000 ug/m^3, field evaluation indicates the monitor yields a much higher ambient air value than that obtained by the accepted EPA-TGS-ANSA method. The effect of temperature and sunlight on the monitor is minimal and other common pollutants including nitric oxide do not affect the response of the monitor.

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CONTENTS

Abstract	iii
Figures	vi
Tables	vi
Sections	
1 Introduction	1
2 Conclusions	4
3 Recommendations	5
4 Experimental	6
5 Selection of an Absorber	11
6 Selection of a Membrane	15
7 Evaluation of Method	18
8 Discussion	24
References	25
Inventions	26

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FIGURES

No.		Page
1	Clean Air Preparation Apparatus	7
2	Personal Monitor	9
3	Effect of Temperature on Permeability	16
4	Response Time	17

TABLES

No.		Page
1	Absorber Efficiency	12
2	Stability of Absorbed NO_2 in Thymol-NaOH	14
3	Interference Studies	19
4	Linearity	20
5	Field Evaluation	22
6	Spiked Air Samples	23

SECTION I

INTRODUCTION

GENERAL

There has been an obvious need for reliable methods which can be used for measuring the air pollution levels to which individuals are exposed. Such personal monitoring seriously restricts the type of method that can be used since measuring and/or sampling devices must be considerably smaller and lighter than those used conventionally.

The need for improvement over present methods encouraged us to develop methods for determining the average concentrations of SO_2(1) and CO(2) in the air using a sampling technique based on gas permeation. The sampling step involves placing an absorber on one side of a permeable membrane while the other side is exposed to air. The pollutant to be measured permeates through the membrane at a rate proportional to concentration and reacts or is absorbed by a suitable absorber. The absorbed pollutant or reaction products of the pollutant are then determined by conventional methods and the concentration is calculated using a predetermined calibration constant.

The purpose of this project was to develop a personal monitor for NO_2 based on this unique sampling approach. Sampling by permeation is particularly useful for personal monitoring because hardware for such a monitor can be extremely small and lightweight. Maintenance of a carefully controlled flow rate is not required because the rate of collection is controlled by permeation; consequently, the need for auxilliary air pumps, flow-meters, critical orifices, flow controllers, bubblers and impingers is obviated. Generally these monitors provide a reliable, sensitive and specific response to individual gases by careful selection of absorbers and permeable membranes.

OBJECTIVE

The primary objective of this study was to develop a personal monitor for the determination of the average concentration of NO_2 in the air. The proposed system was to involve sampling by permeation through a

polymer membrane as with the methods previously described for SO_2 and CO. The permeated pollutant was then to be absorbed in an appropriate medium and determined by a suitable analytical procedure. The final prototype of the personal monitor was to be as simple and compact as possible.

PROGRAM APPROACH

Several requirements must be met for the successful development of a method based on the permeation sampling approach. First, an absorber must be found that irreversibly absorbs the gaseous pollutant to yield a stable product. Additionally, this product must be easily measurable and should be formed in an amount proportional to the amount of pollutant absorbed. Next, a membrane must be obtained that is highly permeable to the particular gas to be measured, yet has a permeability which is reasonably independent of climatic variables. Finally, the system must be free of significant interferences.

Initially, this investigation involved a study of various absorbing solutions for nitrogen dioxide. Until recently, this has been an inherent difficulty in methods which have been developed. While alkaline absorbers, such as the sodium hydroxide solution used in the original EPA (Jacobs-Hochheiser method) approach (3,4) have not proved satisfactory, triethanolamine has proved to be an effective absorber (5). Other absorbers such as that used in the Saltzman procedure (6) are efficient but lack desired stability. The first step in this investigation was to examine the efficiency of the conversion of NO_2 to NO_2^- by various absorbers. Two absorbers were studied which gave high absorption efficiency. Continued studies of the stability of absorbed nitrogen dioxide resulted in one absorber being discarded. The effect of temperature, time and exposure to sunlight on the absorbed species was determined because of the effect these parameters would have on the versatility and reliability of the final measuring device. The determination of nitrite was carried out by conventional diazotization and coupling procedures.

After a satisfactory absorbing system had been selected, a thorough study of the permeability properties of silicone rubber was made. Additionally, an interference study was carried out on the common possible interferences. After completion of the laboratory investigations, field evaluation was carried out. Although all former studies indicated that under field conditions the method should work reliably, the field data from the permeation device were about 200 percent higher than that obtained from the TGS-ANSA EPA procedure (7,8). Future studies should be directed to eliminate or find what is causing this discrepancy.

SECTION 7

CONCLUSIONS

A personal monitor for measuring NO_2 concentrations has been developed. It is considerably smaller, lighter and more convenient to use than other equipment available. The unit as described, weighing only 50 grams, could undoubtedly be reduced even further in size and weight. All laboratory evaluations of the developed method indicate that the monitor should work reliably. The method is free of interference from the common pollutants in the air and is essentially unaffected by changing environmental conditions. Unfortunately the field evaluation revealed a discrepancy between the method developed here and an EPA developed procedure. Until this discrepancy is resolved the method should not be used at ambient levels of NO_2. The discrepancy does not invalidate the procedure at the much higher occupational levels.

SECTION 3

RECOMMENDATIONS

Although extensive laboratory evaluations indicated the method developed should work reliably, there was a definite difference between the field data obtained with the permeation device and that obtained with the current EPA procedure. It is recommended that future investigations attempt to determine what caused this discrepancy. This work should include field evaluation at a more conventional sampling site than the one used here. Samples for this work were collected adjacent to a building containing numerous chemical laboratories. It is also recommended that field evaluation of the device be carried out at more conventional occupational levels which are often orders of magnitude larger than ambient levels.

SECTION 4

EXPERIMENTAL

PREPARATION OF NO_2 TEST ATMOSPHERES

The large volumes of air required for these investigations necessitated the use of an ambient air cleanup assembly. Tank air could not conveniently be supplied in required quantities. Air from the laboratory was decontaminated by pumping it over a humidifying solution (see Reagents) through a series of chromate oxidizers and NO_2 sensitized charcoal absorbers (Section 1), and finally over activated charcoal (Fig. 1). No detectable amount of NO or NO_2 was found in this air. The air was further dried with silica gel so that permeation tubes used in these investigations would not deteriorate. The humidifying solution was changed daily and the sensitized charcoal absorbers were changed as needed to maintain a NO and NO_2 free air stream. Experience dictated that about 500g of sensitized charcoal was needed per day when air was used at a flow rate of 10 l/min.

Decontaminated air was mixed with dry air that had been passed over selected permeation tubes or through an ozone generator for the preparation of test atmospheres. The test air had an estimated relative humidity of 70 percent and was practically free of CO_2 because the NO_2 absorbers were alkaline. The permeation tubes were prepared in this laboratory by procedures similar to those of other authors (9). Phosphorus pentoxide was placed inside each tube to insure more reliable permeation rates.

REAGENTS

Reagents and chemicals were ACS reagent grade. High quality deionized water was used in preparing all solutions as well as for making up losses due to evaporation.

Alkaline Thymol Absorber--Two tenths gram of thymol (5-methyl-2-isopropylphenol) and 4g of sodium hydroxide were dissolved in 100 ml of water and dilluted to 1 l.

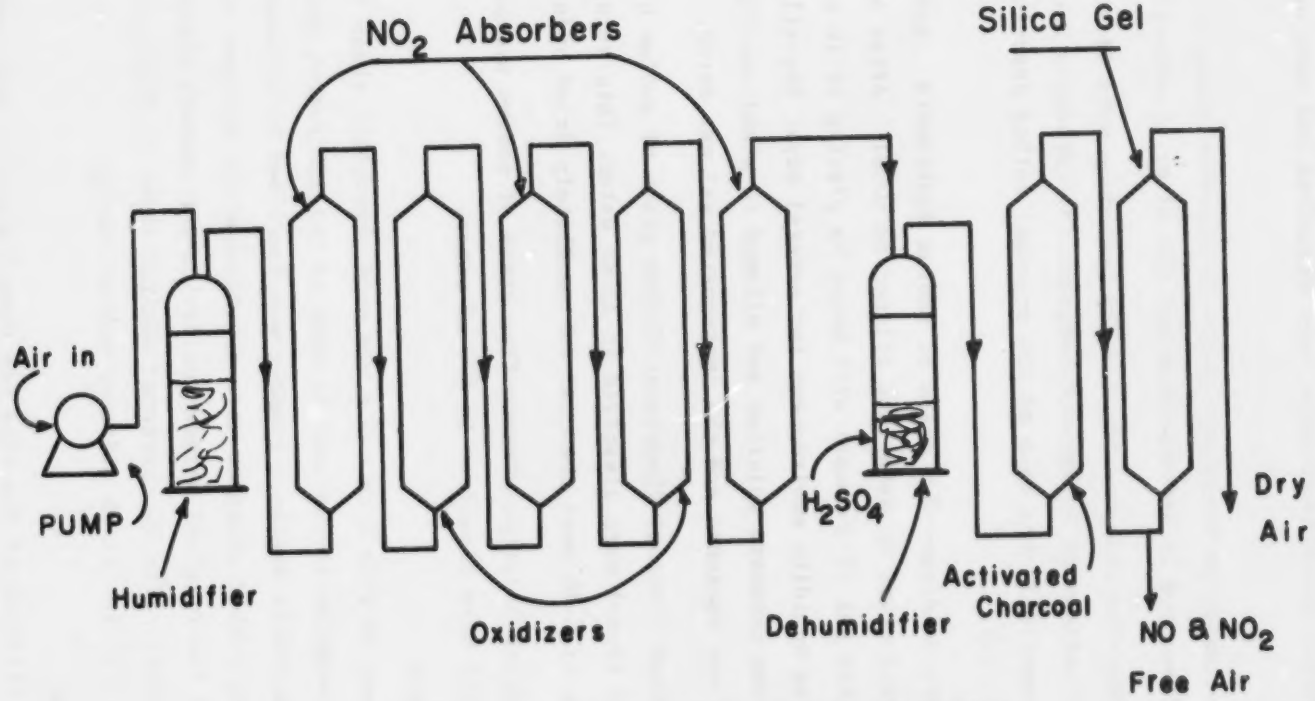

Figure 1. Clean Air Preparation Apparatus

N-(1-napthyl)-ethylenediamine (0.05 percent)--Fifty milligrams of N-(1-napthyl)-ethylenediamine were dissolved and made up to 100 ml with water.

Sulfanilamide in Phosphoric Acid--Thirty-five grams of sulfanilamide were dissolved in 250 ml water and 250 ml of phosphoric acid.

TGS-ANSA--The reagents for the EPA procedure were made up according to specifications of Fuerst and Margeson (8) (appendix D page 37) which are essentially the same as the original method described by Mulik et. al. (7).

Chromate Oxidizer--Ten grams of sodium dichromate and 10ml of sulfuric acid were mixed in water and diluted to 500ml. After saturating 500g of silica gel (8-16 mesh) with water by placing it in a 100 percent relative humidity environment for several days, the silica gel was added to the chromate solution and allowed to stand overnight. The excess liquid was decanted and the impregnated gel was dried.

Sensitized Charcoal Absorbers--Thirty grams of sodium hydroxide and 1.5g of thymol were dissolved in 150ml water. This solution was added to 500g of 6-14 mesh charcoal and immediately dried under vacuum.

Humidifying Solution--Twenty-five grams of sodium hydroxide and 1g of thymol were dissolved in 500 ml water.

APPARATUS

A Beckman DB with 1 cm cells was used throughout these investigations. The permeation device used in most of the laboratory and field tests and a suitable exposure chamber was described by Reiszner and West (1). Neoprene rubber stoppers were substituted for the gum rubber stoppers used in the original permeation device for reasons given elsewhere in this report. The NO_2 personal monitor shown in figure 2 was sealed with a 1/16 in. thick silicone rubber o-ring.

SAMPLING

Ten milliliters of the alkaline thymol absorber were used to charge the permeation device or personal monitor. The monitor was shielded from direct sunlight so as not to exceed more than 1 hour of exposure

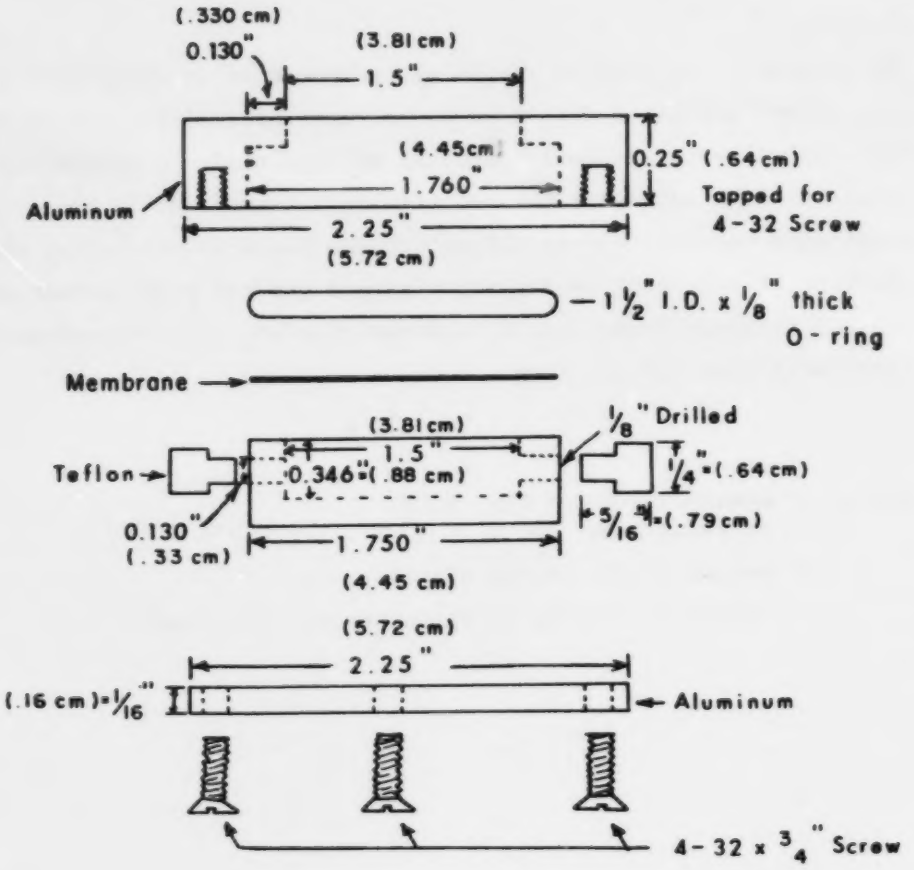

Figure 2. Personal Monitor

per sample period. It was exposed to NO_2 only while attached to the prospective wearer. Normal sampling periods varied from 4 hours to 5 days depending on the sensitivity and type of data required, however, most of the development work was based on sampling times of 24 hours or less. Evaporative losses were replaced as required to maintain contact between the liquid and the membrane.

ANALYSIS

The volume of the trapped sample was adjusted to 10 ml by adding an appropriate amount of deionized water. One milliliter each of the N-(1-napthyl)-ethylenediamine and the sulfanilamide in phosphoric acid solutions was added and the solution was stirred for 10 sec. The absorbance was measured at 545 nm between 5 and 40 min. after the addition of the color developing reagents and the total amount of NO_2 in the solution was found from a calibration plot. The concentration was calculated from the equation:

$$C = \frac{w\,k}{t} \qquad (1)$$

where C = concentration of NO_2, ug/m^3
t = sampling time, hr
w = amount of NO_2^- in the absorber, ug
k = constant, usually in the order of 4000 hr/m^3.

SECTION 5

SELECTION OF AN ABSORBER

The first step in the development of the NO_2 personal monitor was the selection of the absorber. The alkaline thymol absorber, which was first reported by Nash, (11) was chosen after also considering solutions of triethanolamine, (5) and 0.1N sodium hydroxide. All absorbers were analyzed for NO_2^- by the analysis procedure described in the experimental section. The 0.1N sodium hydroxide solution was tested because it was an inherently simple system, and the problems of collection efficiency (4) associated with this absorber should be less pronounced in permeation sampling techniques because permeation permits much slower absorption than conventional procedures in which air is bubbled through an absorber. This reagent was discarded after reproducibility problems were encountered with its use as the absorbing reagent in the permeation device.

Alternative absorbers were tested after erratic results were obtained which were attributed to the 0.1N sodium hydroxide absorbing reagent. Particular attention was given to the absorption efficiency of the various absorbers shown in Table 1. The efficiency was determined using conventional coarse fritted glass bubblers. The conversion efficiency of NO_2 to NO_2^- was 90 percent for the alkaline thymol (5-methyl-2 isopropylphenol) and 93 percent for quaiacol (o-methoxyphenol) (7) solutions and 87 percent for triethanolamine. The data for guaiacol (7) and triethanolamine (5) solutions correlated well with those reported by other observers using similar absorbing systems. Although all three absorbers worked well, the thymol absorber was chosen because guaiacol displayed a slight positive interference with O_3 due to the formation of a colored oxidation product and triethanolamine blanks were found to increase with time. Of particular significance was the fact that the alkaline thymol solution was essentially non-toxic and, therefore, would not endanger the wearer of a personal monitor.

The effect of environmental conditions that a permeation device would be exposed to were also considered in the selection of an absorbing

Table 1. ABSORBER EFFICIENCY

Composition	Percentage Conversion of NO_2 to NO_2^-*	
Sodium hydroxide	(15-70)	(4)
Triethanolamine	87 (85)	(5)
Sodium hydroxide and thymol	90	
Triethanolamine, guaiacol and sulfite	(99)	(7)
Guaiacol and sodium hydroxide	90	

*Literature values in parenthesis

reagent. Both guaiacol and thymol solutions darken upon standing in direct sunlight; however, only moderate shielding was necessary to eliminate this effect and no shielding was required indoors. Exposure of the thymol absorber to direct sunlight in a clear pyrex flask for one hour yielded a value of about 0.03 ug/10ml NO_2^- higher than that of the unexposed reagent upon analysis. This corresponded roughly to the detection limit at the 95 percent confidence level of the method and was equivalent to 10 ug/m^3 for the standard permeation device or personal monitor. Therefore, it was considered unlikely to have any significant effect from sunlight exposure unless the monitor was exposed to sunlight for several hours because the person wearing the monitor and the monitor itself will act to shield the absorbing reagent from sunlight. Even when the person wearing the monitor faces the sun the membrane will substantially reduce the amount of light transmitted to the reagent.

The study of the effect of environmental conditions on an absorber must naturally include the investigation of the possible loss of NO_2 while the monitor is sampling and during the subsequent storage period before analysis. The losses of absorbed NO_2 (i.e., NO_2^-) over a 29 day period as displayed in Table 2 were negligible when the thymol absorber was maintained at 30°C. The absorber was also effective in trapping NO_2 a month after preparation of the reagent and was efficient after absorbing considerable quantities of CO_2. Thymol dissolved in sodium carbonate was found to be as effieient an absorber as the more alkaline solution containing sodium hydroxide.

Table 2.. STABILITY OF ABSORBED NO_2 IN THYMOL-NaOH

Percentage Loss of NO_2 in 29 days at 30°C	Initial Concentration ug/10ml solution
16	0.5
3	2.0
3	4.0
3	8.0
3	16.0
3	32.0

SECTION 6

SELECTION OF A MEMBRANE

The choice of a permeable membrane was of utmost importance to the successful development of a personal monitor. The membrane must be highly permeable to NO_2, yet the permeability must be independent of climatic variables. The necessity of high permeability required that a silicone membrane be used, however, several different types were available. The first choice was the dimethyl silicone rubber that was used in the development of the SO_2 permeation device (1), because it is in general the most permeable of the silicone rubbers. Fortunately no further examination of membranes was necessary because the rate of permeation was sufficiently high and essentially free of temperature effects. There was only 0.25 percent change in the permeation rate for a $1^{\circ}C$ change in temperature when the device was operated over the range from $0^{\circ}C$ to $50^{\circ}C$ (Fig. 3). This is less than one would expect with most battery operated pump samplers. A personal monitor with an 11 cm^2 membrane was found to detect as little as 10 ug/m^3 for a 24 hour sampling period or 30 ug/m^3 for an 8 hr. sampling period.

It is interesting to note that permeation starts almost immediately after exposure to NO_2 as evidenced in Figure 4. In this experiment the exposed sample was removed and the permeation device was recharged with unexposed reagent every 10 min. Obviously the permeation rate exceeds 90 percent of the equilibrium rate in the first 10 min.

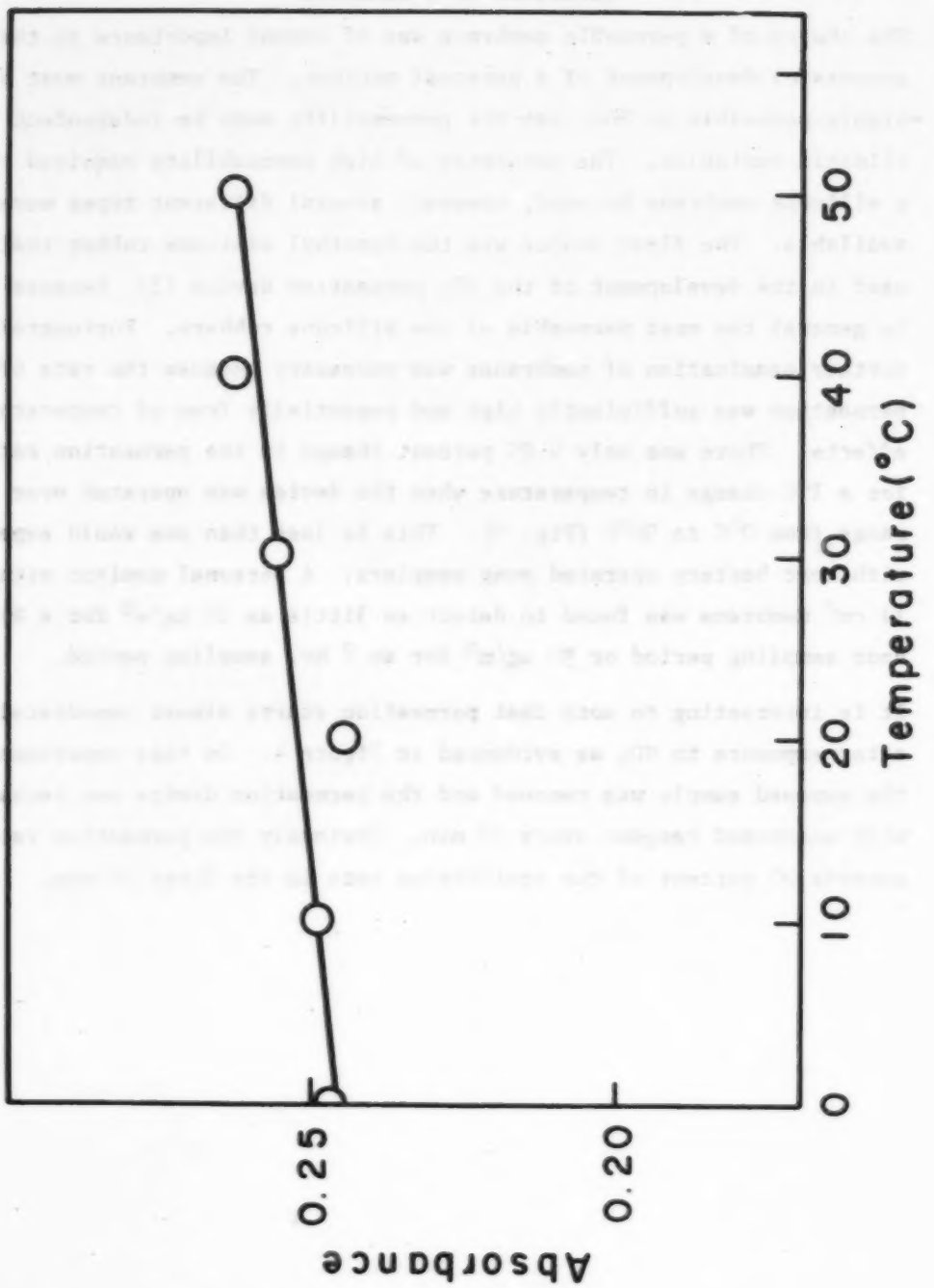

Figure 3. Effect of Temperature on Permeability

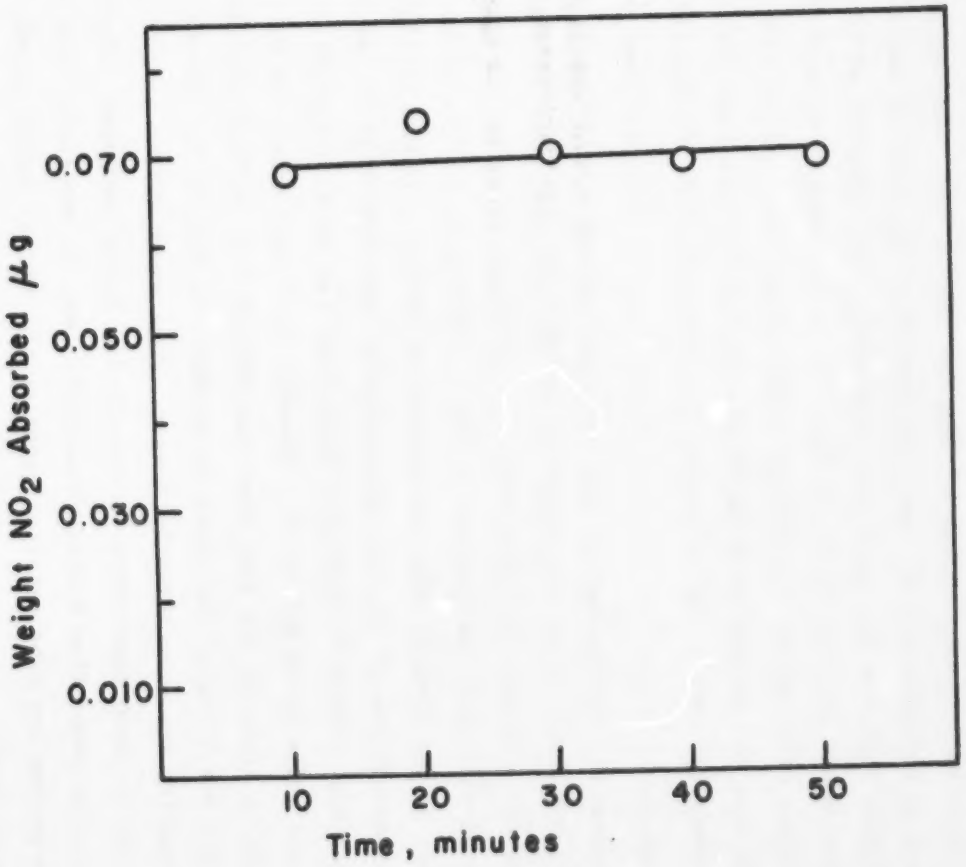

Figure 4. Response Time

SECTION 7

EVALUATION OF METHOD

INTERFERENCE STUDY

The data from the interference studies are presented in Table III. As can be seen there was essentially no interference from relatively high concentrations of SO_2, O_3, NH_3 or H_2S. Although there was an interference at concentrations of NO an order of magnitude higher than the NO_2 level, no interference was observed at NO levels that would normally occur in air. It is interesting to note that the effect of NO on the permeation device at a concentration 1000 ug/m^3 NO_2 was the maximum that would be expected from the fact that the permeation rate of NO is roughly 5 percent of that for NO_2 (12).

LINEARITY

Generally, the permeation rate of a gas through a membrane is directly proportional to the concentration of that gas over many orders of magnitude change in concentration; therefore, sampling arrangements based on permeation respond linearly. During an early portion of the work it was thought that the permeation device response to high concentrations of NO_2 was substantially less than that at low concentrations. Table 4 indicates that there may be an effect at concentrations below about 200 ug/m^3. However, this conclusion can hardly be made in light of the fact that the estimated 95 percent confidence limit was 11 ug/m^3 for these determinations and the deviations from linearity are only slightly larger. From about 250 to 10,000 ug/m^3 it can be seen that there is essentially linear response. The data from the permeation device measurements may be correlated with either the permeation tube emission rates or the EPA procedure to make these conclusions.

The earlier response to low concentrations of NO_2 was attributed to problems with the testing procedures. One of the first difficulties that was encountered was our inability to free laboratory air of NO and NO_2. The cleanup was finally accomplished by the series of oxidizers and absorbers described in Section 4. In order to realize low

Table 3... INTERFERENCE STUDIES[a]

		NO_2 ug/m³		
Pollutant	ug/m³	Taken	Found[b]	Error%
SO_2	2500	100	107	+7
O_3	500	100	105	+5
NH_3	1200	100	106	+6
H_2S	1700	100	87	-13
NO	7600	876	971	+12
NO	1000	100	153	+53
NO	100	109[c]	116	+6

[a]Twenty hour exposure, average of three determinations.

[b]The standard deviation was 5 ug/m³ at 100 ug/m³ NO_2. The average of three determinations was used here.

[c]Measured by EPA procedure.

Table 4.. LINEARITY

Concentration, ug/m^3		
Taken	Permeation Device	EPA Procedure (8)
38	49	37
78	90	78
139	167	142
258[a]	291	272
498[a]	479	450
1159	1159[b]	1117
3953	4103	N.D.
10,700	10,006	9908

[a]The permeation tube used in these experiments experienced a somewhat variable permeation rate.

[b]Calibration

blank values with the uncontaminated air prepared by this procedure it was found necessary to use freshly soaked and washed neoprene rubber stoppers in the permeation devices. Gum rubber stoppers can cause high and varying blanks (8). Contamination from rubber stoppers does not occur with the personal dosimeter because it uses no rubber seals and rubber does not come in contact with the absorbing reagent.

FIELD EVALUATION

Field testing a method is one of the most important aspects in the development of a method. Unfortunately the method was compared only to the EPA procedure (TGS) and the data are difficult to interpret. Lack of time and equipment made further examination particularly at occupational levels, infeasible. Field evaluation consisted of sampling air that had been drawn from directly outside the laboratory through a single Teflon tube. Simultaneous samples were taken by the permeation procedure and the EPA procedure for periods of about 24 hrs. The data from these studies which are shown in Table 5 indicate a dramatic difference in the two analytical procedures. A reasonable explanation of the higher results obtained with the permeation device is not available at the present time, however, two experiments were designed to characterize this phenomena.

The results of these two experiments are shown in Table 6. Both experiments involved spiking air in the laboratory with known quantities of NO_2. The data indicate that the permeation procedure and the EPA procedure responded to the spike in roughly the same fashion that would be expected by the laboratory evaluations i.e. linearity, etc. This leads one to believe that both methods are working reliably and does not yield an explanation of the field results from the two methods which differ by a factor of about three.

Table 5. FIELD EVALUATION

	Concentration, ug/m^3	
Sampling Date	**Permeation Device**	**EPA Procedure**
7-21	85	36
7-22	112	31
7-24	80	29
7-28	92	36
7-30	67	32
7-31	76	25

Table 6. SPIKED AIR SAMPLES

	Concentration, ug/m^3		Difference, ug/m^3	
	Ambient	Spiked Ambient	Generated	Found
Permeation Device	88	197	85	109
EPA Procedure	27	97	85	70
Permeation Device[a]	82	215	92	133
EPA Procedure	32	134	92	102

[a]The permeation devices were charged with the EPA absorbing reagent. The calibration constant was assumed identical to that obtained with the alkaline thymol absorber.

SECTION 8

DISCUSSION

The permeation method described here can detect as little as 30 ug/m^3 NO_2 based on the 95 percent confidence level of the blank for an 8 hr. sampling period and as little as 10 ug/m^3 for a 24 hr. sampling period. The monitor devised to use this method is extremely light-weight and compact and can be easily worn by personnel in the performance of the most demanding tasks without restricting the wearers movements. The method yields essentially linear response to NO_2 from 40 to 10,000 ug/m^3 and has practically no interference from other common air pollutants. However, there was a marked discrepancy between ambient air levels that were obtained with the new method and the EPA procedure that the method was compared with. Laboratory studies indicated that both methods should work reliably. It is as difficult to believe that the permeation procedure could yield results higher than the true concentration as it is to believe that the EPA procedure could yield low values. This is particularly true in light of the fact that for the permeation device to respond excessively the permeation rate must change or some constituent in the air must be chemically transformed to NO_2^- as it passes through the membrane and is absorbed. A change in permeation rate is inconceivable and the chemical transformation is considered to be unlikely.

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INVENTIONS AND PUBLICATIONS

A. Personal Monitor for NO_2 Using the Permeation Sampling Approach, Reiszner, K. D. and P. W. West, in preparation.

B. The NO_2 personal monitor could be considered an invention.